

MICRO-LEVEL STUDIES ON SELF COMPACTING CONCRETE

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ABSTRACT

Concrete is the most commonly used material for construction. The worldwide production of cement has greatly increased since 1990. Production of cement results in a lot of environmental pollution as it involves the emission of CO₂ gas. Supplementary cementitious materials (SCMs) are finely ground solid materials that are used to replace a portion of the cement in a concrete mixture. These supplementary materials may be naturally occurring, manufactured or manmade waste. Various types of pozzolanic materials that improve cement properties have been used in cement industry for a long time. Metakaolin is a dehydroxylated aluminium silicate. It is an amorphous non-crystallized material, constituted of lamellar particles. From the recent research works using Metakaolin, it is evident that it is a very effective pozzolanic material and it effectively enhances the strength and durability properties of concrete. The research study reported in this paper is on the effects of Metakaolin and class F fly ash on the micro-level properties of SCC at different replacements of cement at various curing periods. The micro-level studies such as Scanning Electron Microscopy (SEM) and Energy Dispersive x-ray Spectrometry (EDS) for 28 and 90 days have been reported. Scanning Electron Microscope (SEM) images indicated that the addition of cement concrete with Metakaolin the formation C-S-H gel in ordinary Portland cement with high performance Superplasticizer cum retarder which was done by finding the various predominant elements present and compounds formed by using EDS analysis. The micro-level properties studied were the micro-crack widths between aggregate and paste, and the chemical elements and atomic Calcium-Silica of the paste near the Interfacial Transition Zone (ITZ). It has been suggested that micro-cracks in the ITZ play an important role in determining not only the hardened properties but also the permeability and durability properties of concrete (Mehta and Monteiro, 2006). Modification of the micro-structure in the ITZ has been one of the greatest concerns since durability and strength of concrete are significantly influenced.

KEYWORDS: SCC, Metakaolin, Flyash, Micro-Structure, SEM, EDS

INTRODUCTION

1. Micro-Level Properties

The addition of mineral admixtures is a successful approach in improving the mechanical properties, permeability and durability properties of SCC. Major constituents of Metakaolin are silica oxide (SiO₂) and alumina oxide (Al₂O₃). Other component includes ferric oxide, calcium oxide, magnesium oxide, potassium oxide, etc. Metakaolin should meet the requirements of ASTM C618 (1993). Fly ash is used as a mineral admixture in concrete and it influences on the micro-structure of concrete (Jing and Stroeve, 2004). In ordinary portland cement concrete, MK reacts with calcium hydroxide in cement paste to form mainly calcium silicate hydrates (C-S-H), C₂ASH₈ (gehlenite hydrate), and C₄AH₁₃ (tetracalcium aluminate hydrate).

The formation of secondary C–S–H by this reaction reduces total porosity and refines the pore structure, thus improving the strength and impermeability of the cementitious matrix.

The comparison of reactivity level of MK and fly ash with controlled concrete (OPC 100%) has been made. The pozzolanic reaction of fly ash reduces the amount of $\text{Ca}(\text{OH})_2$ produced and lowers the Ca/Si ratio of the C–S–H in the cement/fly ash mix (Wesche, 1991). The pozzolanic reaction gives a fly ash concrete having fine pore structure, low permeability, long-term strength gain properties and enhanced durability properties (Sear, 2001).

The Scanning Electron Microscopy (SEM) has been a powerful tool in the examination of cement and concrete micro-structure (Nemati, 1997; Stutzman, 2000). The Energy Dispersive x-ray Spectroscopy (EDS) can be used for spectrum analysis to determine the chemical elements and their peaks along with their relative concentrations (Stutzman, 2000). Metakaolin (MK), which is a relatively new material in the concrete industry, is effective in increasing strength, reducing sulphate attack and improving air-void network. Pozzolanic reactions change the micro-structure of concrete and chemistry of hydration products by consuming the released Calcium Hydroxide (CH) and production of additional Calcium Silicate Hydrate (C–S–H), resulting in an increased strength and reduced porosity. In general, SCMs with higher alumina contents such as MK tend to improve durability (Sanjay *et al.*, 2013). MK is an amorphous pozzolan, with some latent hydraulic properties, that is well-suited for use as SCMs (Bensted, 2002). Kostuch *et al.* (2000) observed that CH was significantly reduced with age for all replacement levels (0, 10, 15 and 20%). Larbi and Bijen (1993) reported that at 100 days of curing, inclusion of MK in mortar decreased pore volume and threshold diameter.

Khatib and Wild (1996) reported that the porosity and pore size distribution of OPC metakaolin (0, 5, 10 and 15%) paste is prepared at a constant water/binder (w/b) ratio of 0.55. Incorporation of Metakaolin in concrete led to refinement of the pore structure. Ambroise *et al.* (1994) determined the total porosity of blended cement with hydration time. Mortars mixtures (binder: sand = 1:3) were prepared: one with plain OPC, and the others with MK blended cements. Poon *et al.* 2001 investigated the porosity and pore size distribution of high performance cement paste blended with Metakaolin. The cement pastes were prepared with 5, 10, and 20% MK contents at w/b ratio of 0.3. Porosity and average pore size of pastes decreased with age. Scanning electron microscopy (SEM) equipped with high-energy dispersive analysis X-ray was used to identify materials present in the cavities, transition zones and cracks to determine failure mechanism of the specimens. SEM is mostly used to examine the micro-structural changes in concrete containing mineral admixtures and it has also been utilized to study the effects of addition of chemical admixtures on the micro-structure of concrete.

2. EXPERIMENTAL STUDY

The term basic micro-structure indicates the structure which develops in concrete at a micro-level when water is added to cement and aggregates. In order to understand the cause, extent and mechanism of deterioration, or how to improve some of the properties of concrete, a thorough awareness of the basic micro-structure of hardened concrete is required. Mechanical properties of concrete mostly depend on its intrinsic micro-structure. The high resolution capability of SEM coupled with the possibility of instantaneous micro-analysis using EDS has opened a world of opportunities in the field of concrete technology which otherwise would have been impossible by the standard petrographic technique using an optical microscope. The micro-structure of concrete is described as an integrated system consisting of (i) hydrated cement paste (HCP); (ii) coarse and fine aggregates and (iii) the interface between aggregate and hydrated cement paste (HCP), also known as the interfacial transition zone (ITZ) or paste-aggregate interface.

Hydrated Cement Paste (HCP)

The hydrated cement paste in a normal concrete with a water/cement ratio (W/C) in the range of 0.50 to 0.55 consists of cement hydration products that include (i) calcium silicate hydrate (C-S-H) which forms 50–60% of the solid volume in HCP; (ii) calcium hydroxide (CH) crystals contributing another 20–25% to the solid volume; and (iii) the remainder is occupied by calcium sulfoaluminate hydrates (ettringite) and monosulfate, Incompletely Hydrated Cement Particles (IHCP), and paste porosity, (small 5–20 nm gel pores accounting for the porosity in solid C-S-H and large capillary pores in the paste matrix). The amount of IHCP in a concrete depends on cement fineness, water/cement ratio, and the degree of cement hydration.

C-S-H Phase

The C-S-H phase is the most important component in concrete since it provides the necessary cementitious or binding property to the final product (by Vander Waals force of attraction) and, hence, contributes to the strength of concrete. Scanning electron microscopy has been extremely useful in characterizing the morphology and chemical composition of the C-S-H phase which can vary somewhat within limits. The C-S-H phase in concrete produced at normal temperature is represented by gel structure whereas, at elevated temperatures, it can range from poorly-crystalline to crystalline. Under the SEM, the overall morphology of normal temperature C-S-H can actually vary from the common fibrous type to irregular grains forming a reticular network. Micro-analysis of C-S-H phase has shown that the Ca/Si molar ratio can vary depending on the water/cement ratio or the presence of mineral admixtures in concrete.

Calcium Hydroxide (CH) Crystals

Calcium hydroxide crystals appear in many different shapes and sizes, starting from massive, platy crystals often tens of microns across with distinctive hexagonal prism morphology, large thin elongated crystals, and blocky masses to finely disseminated crystals. Calcium hydroxide crystals tend to develop a preferred orientation along the paste-aggregate interface with their c-crystallographic axis parallel to the interface (Shondeep L. Sarkar, Xu Aimin, and Dipayan Jana (2001)). In the last few decades, a lot of research has been done regarding the improvement of the concrete performance. Self compacting concrete with high cementitious content, a lower volume and maximum size of coarse aggregate are expected to affect the ITZ in a positive manner, promoting a less porous micro-structure and in improving the hardened properties.

To better understand and correlate the micro and macro-level (mechanical) properties, this investigation is mainly focused on the effect of Metakaolin and class F fly ash on the micro and macro-level properties of SCC after 28, and 90 days. SEM analysis was carried out to examine the micro-cracks width between aggregate and paste and EDS analysis was carried out to determine the chemical elements and atomic Ca/Si ratio of the paste matrix near the ITZ at different ages. These properties were examined on Metakaolin group of mixes such as SCC (100% OPC), MK5, MK10, MK15 and MK20 at different ages, whereas fly ash group mixes like FA10, FA20 and FA30 at different ages (MK15 + FA10), (MK15 + FA10) and (MK15 + FA10) at different ages for the study. The sample preparation and test procedure for determining the micro-level properties of concrete have been described in the section 3.7.5. To carry out SEM and EDS, the samples of approximate size 10mm × 10mm × 5mm were collected from the tested specimens after conducting the compressive strength tests as per IS 516 (1991) on SCC mixes at different ages. Micro-hardness measurements can contribute to characterization of the properties of the ITZ relative to the bulk cement paste matrix and also helps in

estimating the width of the ITZ while reductions in micro-hardness for the ITZ relative to the bulk regions are consistent with the hypothesis and that the ITZ is a region of higher than average porosity, such observations are not universal.

Saito and Kawamura (2006) reported increase in micro-hardness within the aggregate surface, where the ITZ micro-structure is weaker than the bulk region, a depression in the micro-hardness profile is expected within the zone of influence of the aggregate. The profile at the aggregate surface is also being influenced by the strength of the paste aggregate bond. Asbridge *et al.* (2001) determined the variations in the micro-hardness of the hydrated cement matrix as functions of the distance from the aggregate surfaces for specimens in which Portland cement was partially replaced with 0 and 20% of Metakaolin, 0 to 30% of fly ash and combination of MK and FA up to 35%.

3. RESULTS AND DISCUSSIONS

The atomic Ca/Si ratios of SCC for all mixes have been computed based on EDS analysis and are presented in Table 1

Table 1: Atomic Ca/Si Ratio of SCC

S. No.	Designation of Mix	Atomic Ca/Si Ratio	
		28 Days	90 Days
1	SCC (100% OPC)	0.27	0.26
2	MK5	-	0.08
3	MK10	-	0.10
4	MK15	0.13	0.16
5	MK20	-	0.09
6	FA10	-	0.03
7	FA20	-	0.17
8	FA30	0.11	0.16
9	MK15+FA10	0.03	0.15
10	MK10+ FA20	-	0.07
11	MK5+FA30	-	0.20

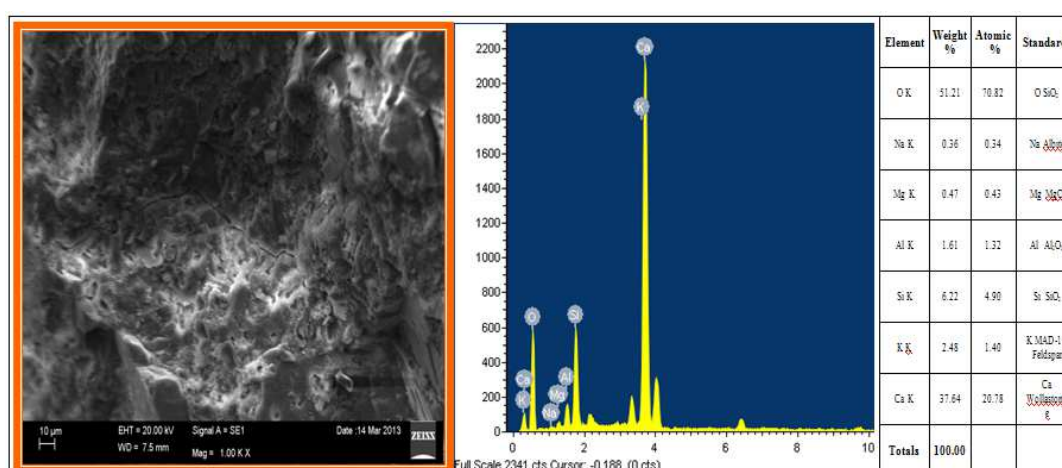


Figure 1: SEM Image & EDS Analysis of SCC (100% OPC) at 28 Days

The SEM image presented in Figure 1 show formation of porous and permeable cement matrix and ITZ on the micro-structure. The EDS analysis of SCC (100% OPC) at 28 days presented in Figure 1 shows that the element SiO₂ is found in large quantity in the concrete followed by Ca, where as the elements such as Albite, MgO, Al₂O₃ and feldspar are found in small quantities.

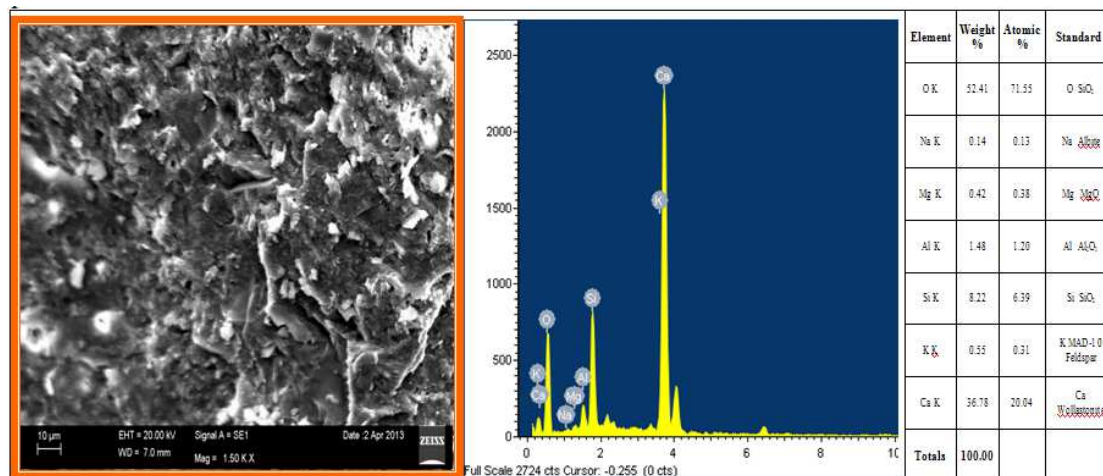


Figure 2: SEM Image & EDS Analysis of SCC (100% OPC) at 90 Days

Figure 2 shows SEM image of SCC (100% OPC) at 90 days. Cracks and large porous and permeable HCP on the micro-structure can be seen from the image. Figure 2 depicts the EDS analysis of SCC (100% OPC) at 90 days. The element SiO₂ present in SCC (100% OPC) at 90 days is more than that of SCC (100% OPC) at 28 days.

Hence, it indicates that the durability property of concrete may be enhanced. The calcium hydroxide is affected due to the hydration of cement. From the Table 1, it is observed that atomic Ca/Si ratio is slightly decreased with the age of concrete which is reflected in the improvement of hardened properties of SCC. This agrees with the findings of Hewlett (2003).

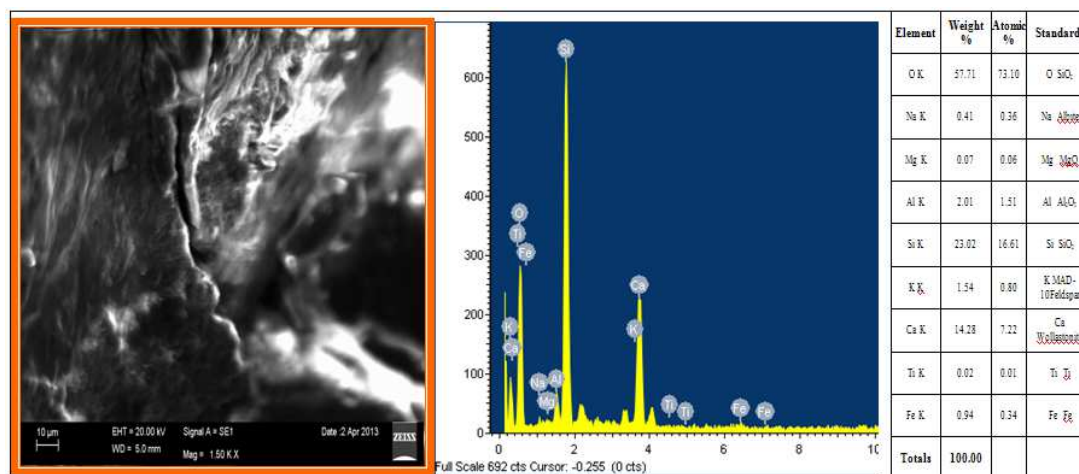


Figure 3: SEM Image & EDS Analysis of MK 5 Mix at 90 Days

The SEM image of MK5 mix at 90 days is presented in Figure 3. The image shows formation of crack and C-S-H phase on the micro-structure. The EDS analysis of MK5 mix at 90 days is exhibited in Figure 3. The atomic Ca/Si ratio is observed to be 0.08 (Table 1).

It is mainly attributed to pozzolanic action of low calcium which reduces the amount of calcium hydroxide Ca(OH)₂ produced during hydration or transforming the calcium hydroxide into C-S-H gel phase. These findings are in concurrence with the observations of Kuroda *et al.* (2000), Wong and Buenfeld (2006), and Xiong *et al.* (2002).

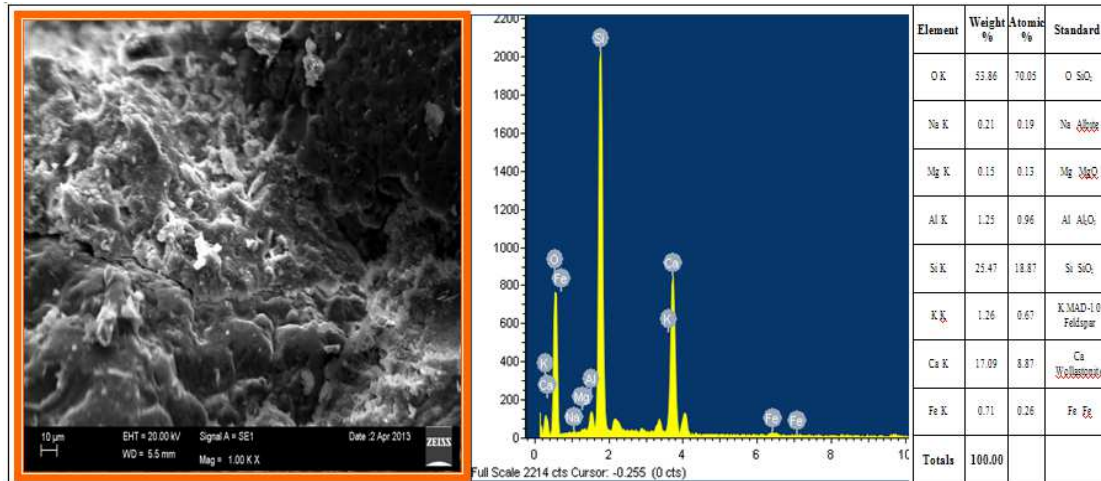


Figure 4: SEM Image & EDS Analysis of MK 10 at 90 Days

The result obtained by SEM image of MK10 at 90 days shown in Figure 4 reveals dense HCP, C-S-H phase and crack on the micro-structure. Figure 4 depicts the EDS analysis of MK10 at 90 days. It can be seen from the Table 1 that the atomic Ca/Si ratio is decreased from 0.26 for SCC (100% OPC) to 0.10 for MK10 mix at 90 days. It is due to pozzolanic action of high reactive Metakaolin.

The partial replacement of cement by Metakaolin is clearly seen in significant reduction of calcium hydroxide peaks with respect to the SCC (100% OPC) which implies that the specimens with Metakaolin produce C-S-H phase on the micro-structure which in-turn increases the hardened and durability properties of concrete.

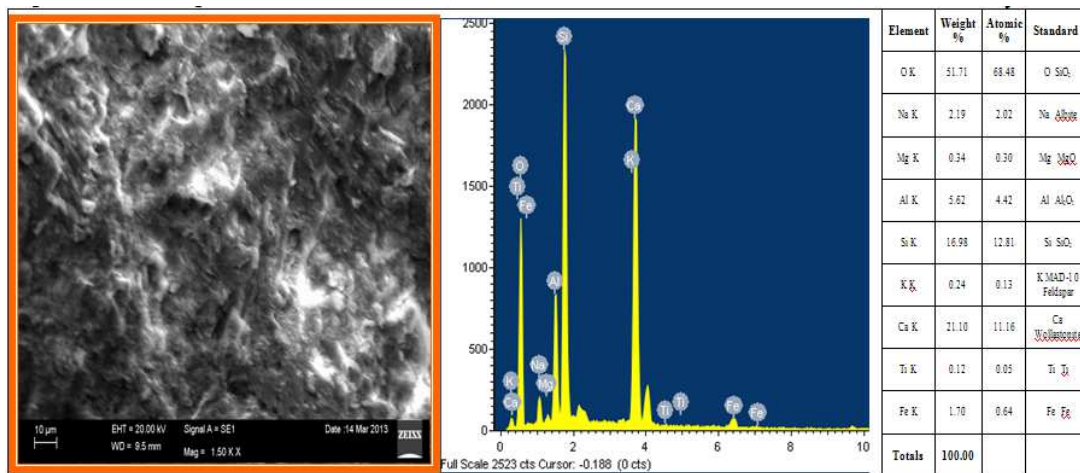


Figure 5: SEM Image & EDS Analysis of MK 15 at 28 Days

Figure 5 depicts the SEM image of MK15 mix at 28 days. Dense HCP, C-S-H phase and porous formation is observed on the micro-structure. ED's analysis of MK15 mix at 28 days is presented in Figure 5. The element SiO₂ is found in large quantity in the concrete followed by Ca. Other elements include Albite, MgO, Al₂O₃, Feldspar, Ti and Fe.

The atomic Ca/Si ratio of SCC for MK15 mix at 28 days reduced to 0.13 from 0.27 for SCC (100% OPC) at 28 days which is due to the presence of high reactive Metakaolin. It results in faster pozzolanic behavior at an early age and produces a more refined concrete than SCC (100% OPC). This behavior is reflected in attaining higher compressive strength of the concrete.

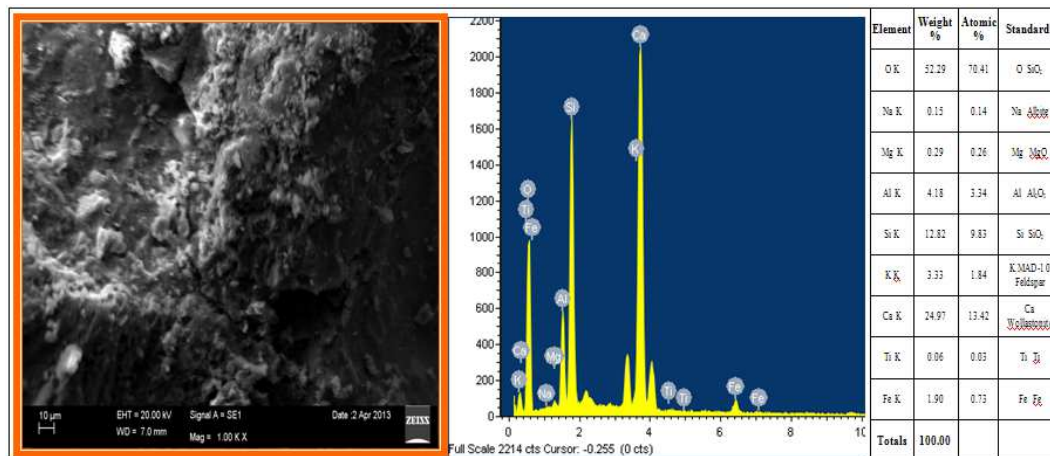


Figure 6: SEM Image & EDS Analysis of MK 15 at 90 Days

The SEM image of MK15 mix at 90 days presented in Figure 6 shows highly dense, C-S-H phase and altering crack on the micro-structure. The EDS analysis of MK 15 mix at 90 days is shown in Figure 6. The element SiO₂ is found in large quantity followed by Ca, whereas the elements like Albite, MgO, Al₂O₃, Feldspar, Ti and Fe are found in small quantities. The atomic Ca/Si ratio found from the Table 1 for MK15 mix at 90 days is lower than that of SCC (100% OPC) at 90 days. It indicates that the addition of Metakaolin resulted in a homogeneous micro-structure characterized by compact and strong C-S-H gel phase. As a consequence ITZ was produced. In addition permeability of concrete of MK15 mix at 90 days observed from RCPT was significantly reduced which reveals good resistance against penetration of chlorides because of the micro-structural densification tortuosity of the pore system.

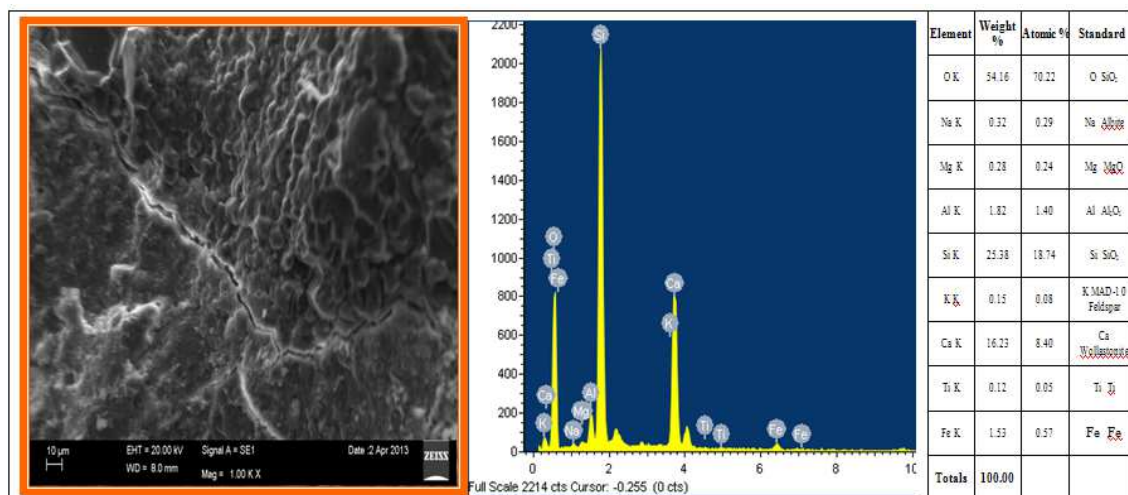


Figure 7: SEM Image & EDS Analysis of MK 20 at 90 Days

Figure 7 depicts the SEM image of MK20 mix at 90 days. Highly dense HCP, strong C-S-H gel phase and reduction in crack width are observed on the micro-structure. The EDS analysis of MK20 mix at 90 days is reported in Figure 7. The various elements along with percentage weight and atomic percent are also tabulated. The element SiO₂ is found in large quantity in the specimen followed by Ca, whereas the elements Albite, MgO, Al₂O₃, Feldspar, Ti and Fe are found in small quantities. It can be seen from Table 1 that Ca/Si ratio is decreased to 0.09 against 0.26 for SCC (100% OPC). The partial replacement of cement with Metakaolin lowers CaO from atomic weight 20.04% to 8.40% which causes quicker densification of the C-S-H gel phase.

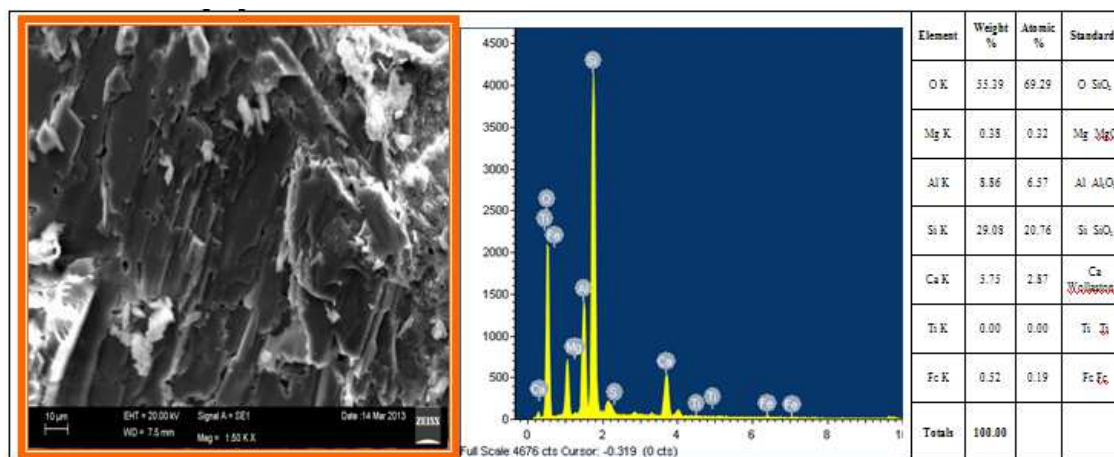


Figure 8: SEM Image & EDS Analysis of FA10 Mix at 90 Days

The SEM image of FA10 mix at 90 days (Figure 8) reveals CH crystals, ettringites and non-fractured surface on the micro-structure. The ettringites can cause internal disruption of HCP. Pores and acicular (needle-shaped) is identified and ettringites are formed on the micro-structure. Figure 8 exhibits the EDS analysis of FA10 mix at 90 days. Higher peak is observed for the element SiO₂, whereas smaller peak is observed for Ca. The other elements such as MgO, Al₂O₃, Feldspar and Fe are found in small quantities.

The atomic Ca/Si ratio as seen from Table 1 is decreased to 0.03 when compared to that of SCC (100% OPC). Partial replacement of cement with fly ash significantly influences the hardened properties of concrete. Its influence is observed on early age gain strength and improved strength gain at later ages.

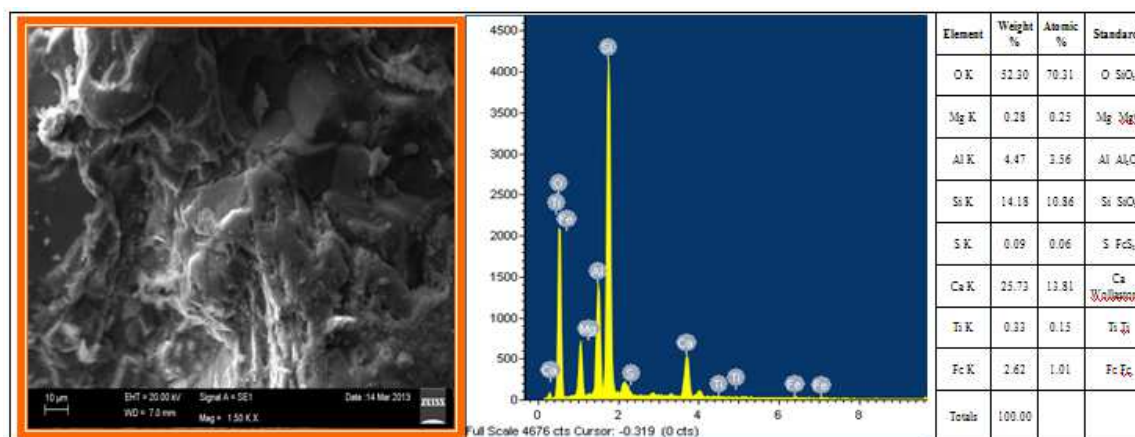


Figure 9: SEM Image & EDS Analysis of FA20 Mix at 90 Days

Figure 9 shows SEM image of FA20 mix at 90 days. The C-S-H phase, cement matrix and aggregate ITZ is observed on the micro-structure. The EDS analysis of FA20 mix at 90 days presented in Figure 9 reveals that the element SiO₂ is a major constituent followed by Ca. The elements like MgO, Al₂O₃, FeS₂, Ti and Fe are minor constituents.

Table 1 shows that the atomic Ca/Si ratio of FA20 at 90 days is lower than of SCC (100% OPC) at 90 days. Although fly ash can enhance the pozzolanic reaction in the early age but initial hydration of fly ash is very slow. The ettringites content and expansion behavior of mixes is correlated gain in strength and dense hydrated cement paste on the micro-structure.

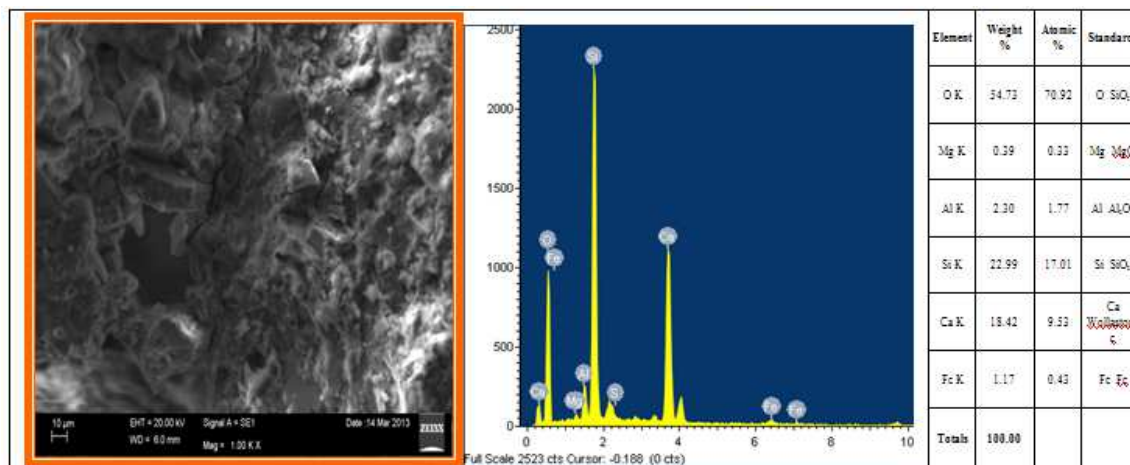


Figure 10: SEM Image & EDS Analysis of FA30 Mix at 28 Days

Figure 10 reports SEM image of FA30 mix at 28 days. The image indicates C-S-H phase, aggregate ITZ and a porous structure on the image. The peaks of various elements obtained from EDS analysis are furnished in Figure 10. The element SiO₂ is present in large quantity in the concrete specimen followed by calcium hydroxide, whereas the elements such as MgO, Al₂O₃ and Fe are found in small quantities.

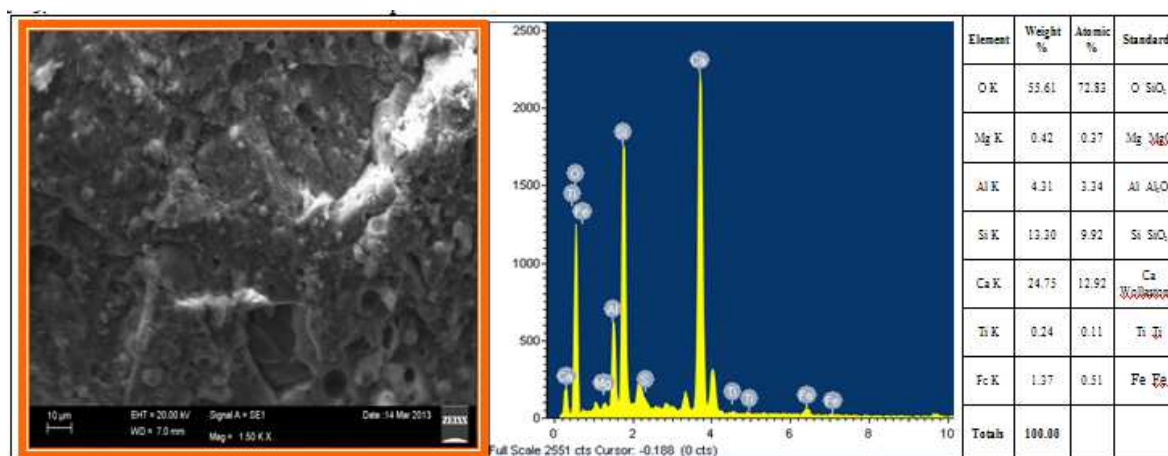


Figure 11: SEM Image & EDS Analysis of FA30 Mix at 90 Days

Figure 11 shows SEM image of FA30 mix at 90 days. The image reveals dense and many spherical particles of fly ash on the micro-structure. Figure 11 presents EDS analysis of FA30 mix at 90 days. The element SiO₂ is found in large quantity in the mix followed by Ca. The other elements like MgO, Al₂O₃, Ti and Fe are found in negligible quantities. The atomic Ca/Si ratios of FA 30 at 28 days is 0.11 and at 90 days 0.16 against 0.27 and 0.26 for SCC (100% OPC). It is mainly attributed to pozzolanic action of low calcium. Fly ash reduces the amount of Ca(OH)₂ produced during hydration or transforms the calcium hydroxide into C-S-H gel phase. Similar observations were made by the researchers Kuroda *et al.* (2000), Wong and Buenfeld (2006), and Xiong *et al.* (2002). Hence, it results in fine pore-structure and causes reduction of micro-crack width in SCC. It also indicates the densification of micro-structure and improvement in the hardened properties of SCC at later ages. The studies already revealed that the hardened properties of fly ash concrete continued to increase with age (Liu, (2010), Siddique, (2003), and Siddique (2011)).

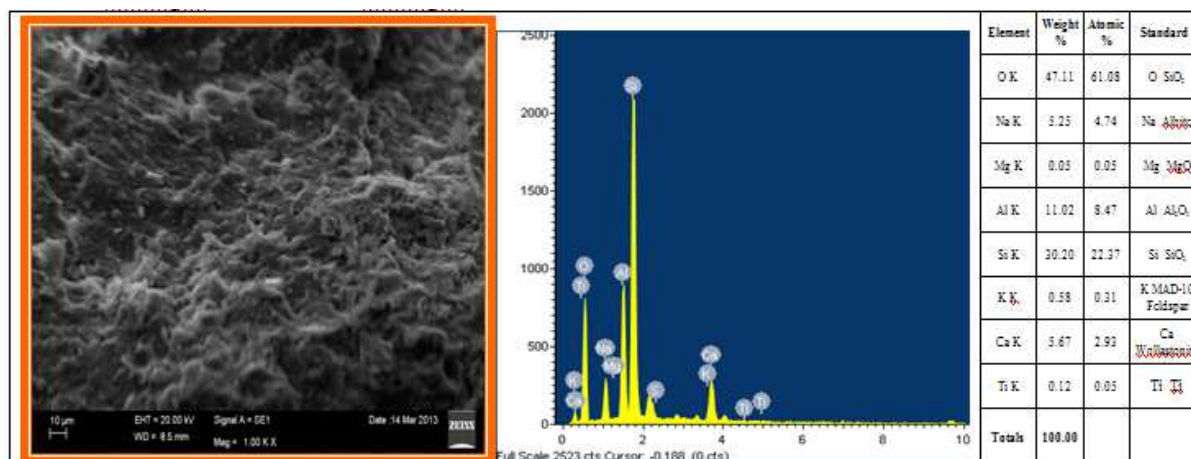


Figure 12: SEM Image & ED's Analysis of MK15+ FA10 Mix at 28 Days

Figure 12 shows SEM image of MK15 + FA10 mix at 28 days. The C-S-H phase, densified, un-fractured surface and small pores are observed on the micro-structure. Figure 12 presents the results of EDS analysis of MK15 + FA10 mix at 28 days. The element SiO₂ is present in large quantity in the specimen followed by Al₂O₃ and Albite. The other elements such as MgO, Feldspar, Ca and Ti are present in small quantities.

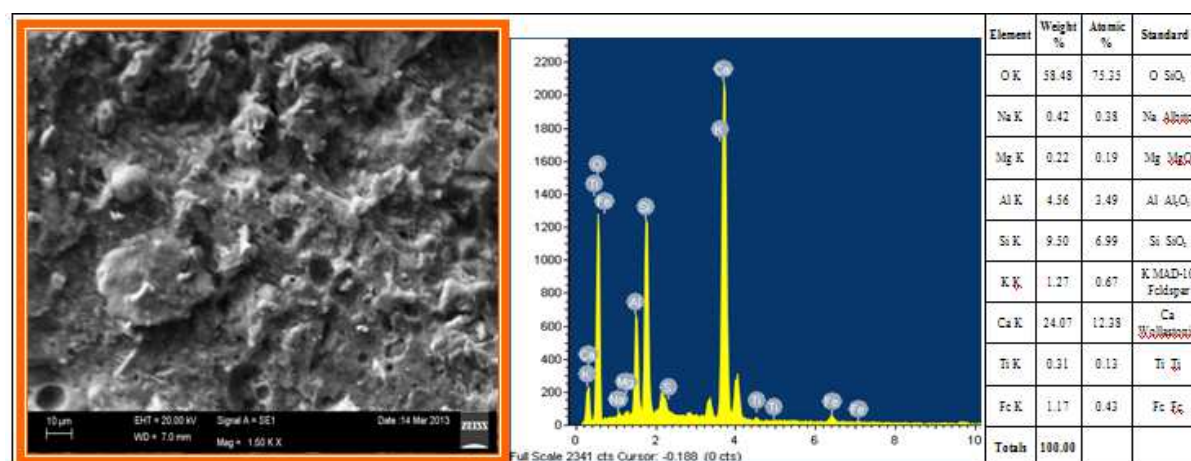


Figure 13: SEM Image & ED's Analysis of MK15+ FA10 Mix at 90 Days

Figure 13 shows SEM image of MK15 + FA10 at 90 days. The image indicates C-S-H phase, spherical shape fly ash particles and ITZ aggregate on the micro-structure. The EDS analysis of MK15 + FA10 at 90 days is shown in Figure 13. The element SiO₂ is the major constituent followed by Ca and Al₂O₃, whereas other elements like Albite, MgO, Feldspar, Ti and Fe are minor constituents. The SEM image of MK 10 + FA20 at 90 days is presented in Figure 14. C-S-H phase, densified fractured surface and large pores on the micro-structure may be observed from the image.

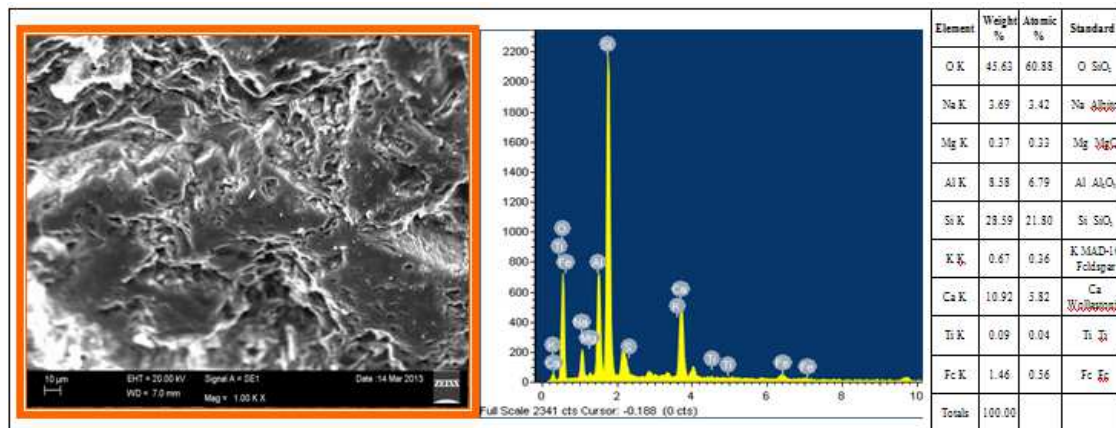


Figure 14: SEM Image & ED's Analysis of MK10+ FA20 Mix at 90 Days

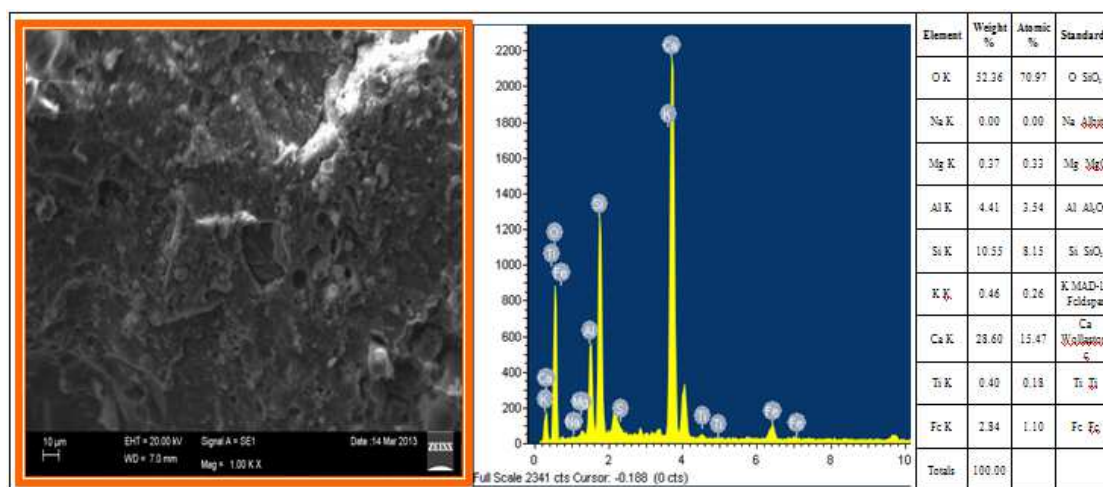


Figure 15: SEM Image & ED's Analysis of MK5+ FA30 Mix at 90 Days

Figure 14 depicts the EDS analysis of MK10 + FA20 mix at 90 days. The element SiO₂ is present in large quantity followed by Al₂O₃ and Ca. Figure 15 represents SEM image of MK5 + FA30 at 90 days. C-S-H phase and spherical shape fly ash a particle in HCP on the micro-structure is found from the image. The EDS analysis of MK5 + FA30 at 90 days shows large peaks for the elements SiO₂ and Ca, whereas for the elements like Albite, MgO, Al₂O₃, Feldspar, Ti and Fe show small peaks (Figure 15). The atomic Ca/Si ratios for MK15 + FA10 at 28 days, MK15 + FA10 at 90 days, MK10 + FA20 at 90 days and MK5 + FA30 at 90 days are 0.03, 0.15, 0.07 and 0.20 respectively. The atomic Ca/Si ratio for SCC (100% OPC) is 0.27 at 28 and 90 days are 0.27 and 0.26 respectively. It shows that the atomic Ca/Si ratios are decreased due to the partial replacement of cement with MK+FA at various proportions. The comparison is made by considering the atomic Ca/Si ratio of SCC (100% OPC) as reference mix. The lower peaks of calcium hydroxide and higher peaks of SiO₂ indicate acceleration in the rate of pozzolanic activity by Metakaolin and fly ash contributing to an enhancement of hardened and durability properties. Thus, better correlation is found with respect to both micro-level and macro-level studies made in this investigation on mixes containing Metakaolin and fly ash in various combinations.

4. CONCLUSIONS

The micro-structural analysis of the hardened properties of SCC reveals that the addition of Metakaolin resulted in

a homogeneous micro-structure, characterized by compact and formation of C-S-H gel. As a consequence, a dense ITZ was produced. The addition of Metakaolin and fly ash caused a refinement of the micro-structure (less interconnected and finer pore structure) and induced the precipitation of small sized C-S-H gel.

Improvement of the micro-structure resulted in higher hardened properties (Compressive strength, splitting tensile strength, modulus of elasticity and flexural strength) due to the fact that pozzolanic gel structure shows better hardened properties than the controlled concrete. In addition, permeability of concrete was significantly reduced (good resistance against the penetration of chlorides) because of the micro-structural densification and increased tortuosity of the micro-structure.

The high reactivity and faster pozzolanic behavior of the Met kaolin particles at early age produced a more refined micro-structure than the SCC (100% OPC). The higher reactivity and agglomerated state appears to be the reason for the higher compressive strength, splitting tensile strength, modulus of elasticity and flexural strengths.

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